The undersigned thanks the Examiner for the courtesies extended during the telephone interview of May 5, 2006.

The objection of claim 17 is now moot as claim 17 has been canceled.

Claims 16-21 were rejected as being obvious over Chen. This rejection is respectfully traversed.

Chen discloses a magnetic recording medium having, *in this order*, an aluminum substrate, a plated NiP layer of thickness in the range of 10 to 20 microns, a sputtered NiP layer of thickness in the range of 5-200 nm and wherein the phosphorous content "should be in the range of 5 to 30 weight percent" (see column 6, line 11 of Chen), magnetic layer and a sputtered carbon overcoat. See Figure 1 of Chen.

On the other hand, claim 16 recites:

> A magnetic recording medium comprising, *in this order*:
>
> (a) a non-magnetic substrate,
>
> (b) a composite nickel coating comprising a sputter deposited nickel layer and an electrolessly deposited nickel layer, the composite nickel coating having a bottom surface contacting the non-magnetic substrate and a top surface, and
>
> (c) a magnetic recording layer on the top surface of the composite nickel coating,
>
> wherein the top surface of the composite nickel coating is a non-polished surface and has a surface roughness (Ra) of less than about 10 Å with the magnetic recording layer thereon, wherein the surface roughness (Ra) is averaged over the entire surface of the top surface of the composite nickel coating.

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In short, at least one distinction between the recording medium of Chen and of this invention is that the sputtered and plated nickel-containing layers are in the *reverse* order. Furthermore, Chen does *not* suggest that one should reverse the order of the two NiP layers of Chen. In fact, in column 8, lines 10-19, Chen states:

> It will be appreciated that nickel-phosphorus layer 12 is either amorphous (i.e., non-crystalline) or near amorphous, and therefore *masks* nonuniformities in the morphology of underlying plated nickel-phosphorus layer 11. Because of this, nickel-phosphorus layer 12 serves as an amorphous uniform nucleation surface for growing sputtered Co-Ni-Pt alloy 13 because there is no crystal structure in layer 12 which alloy 13 tends to emulate. Therefore, magnetic layer 13 exhibits uniform magnetic characteristics. [Emphasis added.]

In Chen's media, the nonuniformities in the morphology of the underlying plated nickel-phosphorus layer 11 are masked by the sputtered deposited layer 12. In order to mask the plated NiP layer 11, the sputtered layer 12 *must* be on top of the plated NiP layer 11, not underneath. Thus, there is simply no suggestion in Chen to arrive at the claimed structure in which the sputtered nickel-containing layer does not mask the plated nickel-containing as the sputtered nickel-containing layer of the claimed invention is beneath the plated nickel-containing layer.

The Examiner has argued that the terms "sputter deposited" and "electrolessly deposited" in the limitations "a sputter deposited nickel layer" and "an electrolessly deposited nickel layer" are simply process limitations that do not distinguish the present claims. Applicants respectfully submit that this is totally incorrect. Applicants respectfully submit that the Examiner's position is similar to arguing that there is no distinction between a fried potato and a baked potato as they are both potatoes and the terms "fried" and "baked" are simply process limitations that define how the fried and baked potatoes were made. Clearly, anybody who has eaten fried and baked

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potatoes would know that this argument is totally illogical as a fried potato has a totally different texture and taste than a baked potato. Similarly, Applicants respectfully submit that "a sputter deposited nickel layer" and "an electrolessly deposited nickel layer" define two *different* types of nickel layers.

In fact, even Chen uses the terms "plated NiP" and "sputtered NiP" to describe the two NiP layers of Chen as these terms are well-known in the art and define two *different* types of layers of NiP having totally different morphology, texture and composition. As explained in paragraph [0006] of the specification, an electroless NiP layer "exhibits desirable physical and chemical properties such as hardness, lubricity, appearance, and corrosion resistance" but it exhibits "nonuniformities in the morphology" (see column 8, lines 12-13, of Chen), while a sputtered NiP layer is "either amorphous (i.e., non-crystalline) or near amorphous" and has "*no crystal structure*" (see Chen, column 8, lines 11-12 and 16-17; also see Chen, column 8, lines 23-33; emphasis added). On the other hand, persons of ordinary skill in the art would recognize that the electroless NiP layer "exhibits desirable physical and chemical properties such as hardness, lubricity, appearance, and corrosion resistance" but it exhibits "nonuniformities in the morphology" because "electroless nickel deposit exhibits a micro crystalline Ni (1,1,1), structure by X-Ray analysis" as explained on page 4, first line under the section "STRUCTURE" of the attached document entitled "Technical Notes: Electroless Nickel Properties," which is available on the Internet at www.stapletontech.com/pdf_files/State_of_EN_Science.pdf. In short, persons of ordinary skill in the art could easily distinguish between "a sputter deposited nickel layer" and "an electrolessly deposited nickel layer" and recognize that they define two *different* types of nickel layers.

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The Examiner has also argued that "Chen et al. teach that it is preferred in their invention to provide a NiP surface that is uniform (i.e. smooth) in order to create a magnetic recording layer having a uniform crystalline structure (see col. 5, lines 53-57 and col. 6, lines 14-28)." See page 3, lines 6-9, of the Action. A careful examination of Chen, in particular, col. 5, lines 53-57 and col. 6, lines 14-28, shows that *nowhere* does Chen disclose a "smooth" NiP surface. The problem that Chen attempts to solve is "nonuniform magnetic characteristics ... [due to] nonuniformity of the crystal structure or morphology of the underlying substrate in the case of crystalline substrates." See Chen, column 5, lines 2-4. Thus, Chen "is silent with respect to the specific values of Ra" as acknowledged by the Examiner, but furthermore there is *nothing* in Chen to lead a person of ordinary skill to conclude that "the reference is suggestive of a range of Ra that is as low as 0Δ." Please see page 3, first full paragraph of the Action.

On page 3, second full paragraph, the Action states that "the examiner takes that position that Chen et al. discloses a non-polished NiP layer because it teaches a method of forming the structure that does not include a polishing step in between deposition of the second NiP layer and the overlying magnetic layer." Applicants respectfully submit that the Examiner is incorrect. In column 4, lines 67-61, Chen states that "substrate surfaces are typically polished and textured prior to magnetic film deposition to provide aerodynamic characteristics which enable the read/write head to fly." Subsequently, Chen does *not* state that the specific process of Chen is *not* "typical" such that Chen "does not include a polishing step in between deposition of the second NiP layer and the overlying magnetic layer" as assumed by the Examiner. Clearly, in the totality of Chen's disclosure, it is totally incorrect for the Examiner to assume that Chen "does not include a polishing step in between deposition of the second NiP layer and the overlying magnetic layer."

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In light of this Amendment, a Notice of Allowance is respectfully solicited.

In the event that the transmittal letter is separated from this document and the Patent and

Trademark Office determines that an extension and/or other relief is required, applicants petition

for any required relief including extensions of time and authorize the Commissioner to charge the

cost of such petitions and/or other fees due in connection with the filing of this document to

Deposit Account No. 03-1952 referencing attorney docket no. **146712011100.**

Respectfully submitted,

Dated: May 9, 2006   By: _____

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Serial No. 10/775,712
Docket No. 146712011100

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ELECTROLESS NICKEL DEPOSIT PROPERTIES

Photo of Electroless Nickel

GENERAL

Electroless nickel coatings have been available to engineers and designers for about forty years. This process has been called autocatalytic, chemical nickel plating, or electroless nickel.

The basic chemistry involves several simultaneous reactions which cause nickel ions, in an aqueous solution, to be reduced to nickel metal. This reaction proceeds without the use of externally applied electrical potential, therefore electroless.

The coating produced is a uniform and hard alloy of nickel and phosphorus. It is applied over the various metals and substrates to provide corrosion and wear resistance.

This paper is intended to provide an overview into the physical and chemical properties of the deposit's, present applications, as well as the test methods available for evaluating the deposit. This document covers only those produced by reduction of nickel by sodium hypophosphite, however a brief description of the other systems is included.

ELECTROLESS NICKEL PROCESS

There are several different electroless nickel deposits which can be produced by reduction from aqueous solutions of nickel. These processes produce a wide range of deposit properties. The different types of deposits offers the design engineer the ability to tailor

the physical properties and achieve the greater performance in the application. Some of the desired properties include increased as plated hardness, improved corrosion resistance, increased wear, better lubricity, wire bondability, solderability, and magnetic and electrical properties. These different deposit types are generally produced from specific solutions which have been formulated for the specific property.

Electroless nickel plating solutions operate by using the laws of electrochemistry, even though external electrical power is not applied. The basic reaction is accomplished when hydrogen is released by the reducing agent (sodium hypophosphite), oxidizing it and producing a negative charge on the surface. This reaction will only proceed if a catalyst (part) is present. The charge on the surface causes a layer to form which the nickel passes through and is reduced, releasing hydrogen.

This process continues until the part is removed from the solution. The controlling factors of this chemistry are temperature, pH, nickel, hypophosphite, and trace elements present. A uniform, consistent deposit and plating rate can be produced by controlling and adjusting the chemistry to account for the increase in dissolved solids within the solution.

The deposits covered in this document were produced from solutions operated above 180(F) and 82(C), with a pH between 4.0 to 5.0. By increasing the hypophosphite, lowering the pH, and selecting specific buffer formulations the amount of phosphorus deposited on the substrate can be increased. Different solutions are more sensitive to these changes, with the range of phosphorus between 0.5% to 13.5% mass weight. The general spread for each solution is 1% phosphorus, with a slight increase for aging of the solution.

Operating an electroless nickel solution requires several chemical additions to maintain the appropriate chemical reaction. Each addition reduces the solubility of the solution, until it is finally saturated. Then chemicals cannot be added without some ingredients precipitating. When this occurs the solution must be discarded or diluted. The amount of chemicals added in relation to the amount of metal deposited represents the efficiency of the system. By reducing the concentration of the solution the number of 100% metal turnovers may be increased. However, the ratio of chemicals used to amount of metal deposited is proportionally consistent for each plating system.

ELECTROLESS DISPERSION COATINGS

There is a class of electroless nickel deposits which contain hard particles or lubricant particles. These deposits are produced from solutions with low concentrations of phosphorus or boron, and have micro-sized particles like PTFE and Silicon nitride $(Si_3(NO_2)^4)$, Silicon carbide (SiC) and other inclusive elements. These deposits provide particular, unique wear resistance and offer increased serviceability. There primary use has been in aggressive abrasion and erosion wear applications. Stapleton Technologies provides these coatings in the TRIBOL processes.

COMPOSITION

Electroless nickel deposits are alloys comprised primarily of nickel and phosphorus with several minor constituent elements. The range of phosphorus is between 0.5% and 13.5% by mass, and the balance is nickel. Many methods have been used to measure the phosphorus in the deposit with Inductively Coupled Plasma (ICP) and EDX being the most widely used and generally the most accurate.

The following table was assimilated from an ICP analysis of deposit samples produced by varying temperatures and pH levels of our AUTONIC MB, MX, and HX series processes.

Care in processing and monitoring of conditions and process chemistry will insure consistent alloys are produced. For some food and medical applications certain elements may be restricted, therefore controls must be maintained.

MID & HIGH PHOSPHOROUS DEPOSIT TYPES

PROCESS CONSTRAINTS								
	AUTONIC MB			AUTONIC MX			AUTONIC HX	
Test Runs A1	A2	A3	B1	B2	B3	C1	C2	C3
pH 4.4	4.8	4.6	4.4	4.8	4.6	4.4	4.7	4.6
Temp. (C) 90	90	90	90	90	90	90	90	90
NaH_2PO_2 35	35	20	40	35	20	35	35	20

ALLOY ELEMENTS								
Nickel	88.4	89.9	94.13	86.7	89.9	94.6	87.1	88.6	90.27
Phosphorus	10.6	9.4	5.3	12.6	9.4	4.91	11.8	10.4	9.52
Aluminum	.0004	.0004	.0004	.0001	.0007	.0001	.0019	.0017	.0017
Cadmium	.0003	.0001	.0001	.0001	.0001	.0001	.0001	.0001	.0001
Chromium	.002	.0015	.0019	.0014	.0015	.0015	.0014	.0017	.0014
Copper	.0061	.0001	.0002	.0015	.0001	.0001	.0013	.0131	.007
Iron	.0002	.00006	.0001	.0006	.0006	.00004	.0003	.0005	.0001
Lead	.0030	.0022	.0026	.0027	.0022	.0026	.0021	.0029	.00052
Zinc	.0036	.0023	.0025	.0027	.0023	.0021	.0024	.0036	.0029

Table 1. Alloy Composition of Electroless Nickel

The alloy produced is dependent upon the chemistry of the solution, the kinetics of each reaction taking place, and the operating conditions. Studies have shown that certain elements incorporated into the deposit and certain stabilizers effect the structure and ultimately effect the corrosion and wear resistance. Elements like iron, chromium, lead, selenium, mercury, bismuth, and antimony have been linked to problems in corrosion

performance and internal stress. By using specific bath formulations, solution constraints, and special pretreatments, these elements can be controlled and their effect minimized.

STRUCTURE

An electroless nickel deposit exhibits a micro crystalline Ni (1,1,1), structure by X-Ray analysis in the as plated condition. Metalographic studies of the deposit show a lamellar structure parallel to the surface of the deposit. The lamellae structure is made visible by anodic etching in a chromic acid solution.

It is thought the lamellae are variations in phosphorus content and/or nickel in the alpha and beta phases. These lamellae are separated by a few micrometers, and appear to be a result of the nature in which the chemical reduction reactions proceed. It is thought the phosphorus is maintained at nearly 15% in the deposit at the interface. As the reaction proceeds a periodic shift in the equilibrium occurs and a thin layer of phosphorus is trapped in a diffusion restricted zone. As the deposit grows this moving wave of phosphorus diffuses towards the surface, leaving this thin layer.

Deposits which are low in phosphorus will have a significant galvanic current on the surface and subsequent etching will make the lamellae visible, while higher phosphorus coatings will be much more difficult to etch.
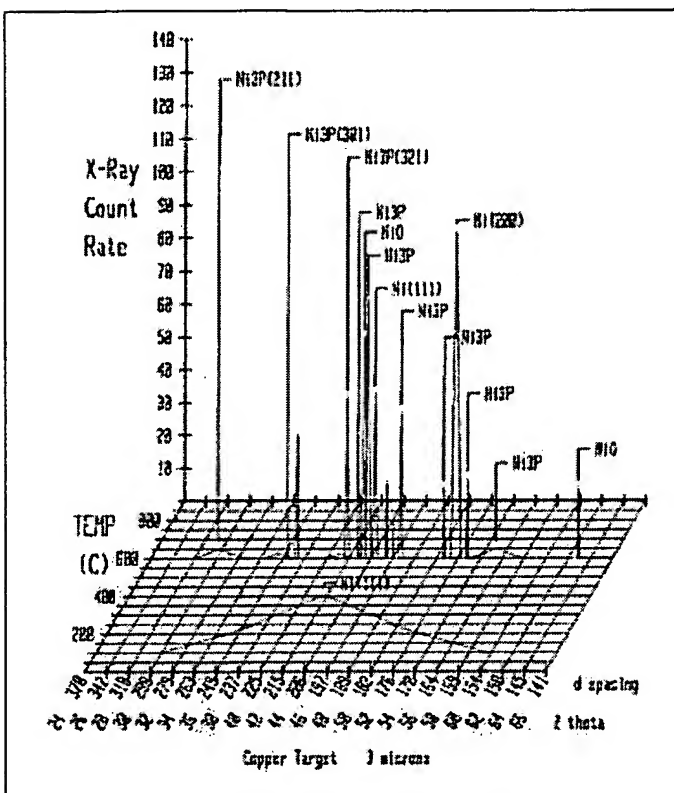

Figure 1 SEM of Surface

Figure 2X -Ray Defraction Analysis

Several researchers have described and studied the relationship of nickel and phosphorus within the deposits. We offer some of their theories and concepts.

A.W. Goldenstein, W. Rostoker, F. Schossberger and G. Gutzeit studied and reported, in 1959, the 7% to 10% P coatings had amorphous, super saturated solution of P in non-crystalline nickel structure, with a disorder similar to that of liquid. Goldstein, A.W.; Rostoker, W.; Schossberger, W. and Gutzeit, C., "Jnl Electrochemical Society" 104 (1959).

The concept of amorphous structure was refined by V.P. Moissejev in putting forth a three phase system: beta nickel less than 5% phosphorus by mass; alpha and beta nickel between 5% and 8.5% mass; and alpha nickel phase with a phosphorus content of 8.5% to 15% mass.Missejev, V.P.; Isvestija, AN USSR, Phys. 26 (1962) 3.378

A divergent view was proposed by A.H. Graham, R.W. Lindsey, and H.J. Read, which stated that a super saturated solid solution of 7% to 9% phosphorus and crystalline and fibrous was formed on copper substrates. This deposit appeared crystalline and fibrous and had the preferred orientation of nickel (1,1,1).

Graham, A.H.; Lindsay, R.W.; Read, H.J., "Jnl Electrochem Society" 109, (1963) 12, 1200, 112 (1965) 4, 401.

J.P. Randin H reported that he found Ni2P in the as plated deposit with very little free phosphorus. Randin, J.P.; Hintermann, H.E., "Jnl Electrochemical Society", 117 (1970) 2, 160.

This condition will occur at high phosphorus levels after heat treatment, but seems quite foreign for the as plated condition.

Most of the researchers have reported that heated deposits will have a grain size of nickel phosphide crystallites in the range of 15 A to 100 A, with a typical 20 A to 60 A   Pai, S.T.; Marton, J.P. and Brown, J., "Jnl Applied Physics", 43 (1972 282.

It is theorized that extremely small grain sized deposits are produced by some cluster free high phosphorus deposits and that standard X-ray analysis with copper targets can not produce the diffraction.

When an electroless nickel deposit, Ni (1,1,1), is heated above 200 (C) crystallization begins causing nickel to precipitate nickel phosphides. Studies by Stapleton Technologies show many different forms of nickel phosphide are produced over a broad heat treatment. These nickel phosphides take the form of Ni3P (211), (301), (321), along with several other structures.

Crystallization of the phosphorus nickel deposit causes the coating to harden and shrink. This changes the volume and ductility, as well as other properties. This result can be used to produce a more wear resistant surface. However, micro-cracking may occur and degrade the corrosion performance of the coating.

When heating immediately after deposition, an equilibrium of phosphorus is produced at the interface. With prolonged high temperature heat treatment, the phosphorus near the interface of the substrate will diffuse, leaving a lower phosphorus coating in that region. A nickel ion intermetallic layer will also be produced with high heat treatment. This condition is helpful in obtaining adhesion on titanium and other passive, oxidizing metals.

The degree of precipitation depends upon time and temperature of the of heat treatment these in turn will effect hardness, magnetics, electrical properties, elasticity, tensile strength and various other properties.

NICKEL PHASES

The nickel phosphorus alloy contains nickel with a solid solution of phosphorus in the as-plated condition. These as plated deposits have a proffered orientation depending on the phosphorus content. By heating the deposit a transformation to a stable equilibrium is achieved. In the graph below the results of several researchers including Konstantiov, Hansen, Koeneman and Metcalfe shows the phases which are in equilibrium. The B phase consists of a crystalline solid solution of phosphorus in nickel. The Y (gamma) phase is totally amorphous and exists between 10.5 and 15% phosphorus. Duncan, R. N. "The Metallurgical Structure of Electroless Nickel Deposits: Its Effect on the Coatings' Properties" Product Finishing, EN93 Proceedings, November 10-12, 1993
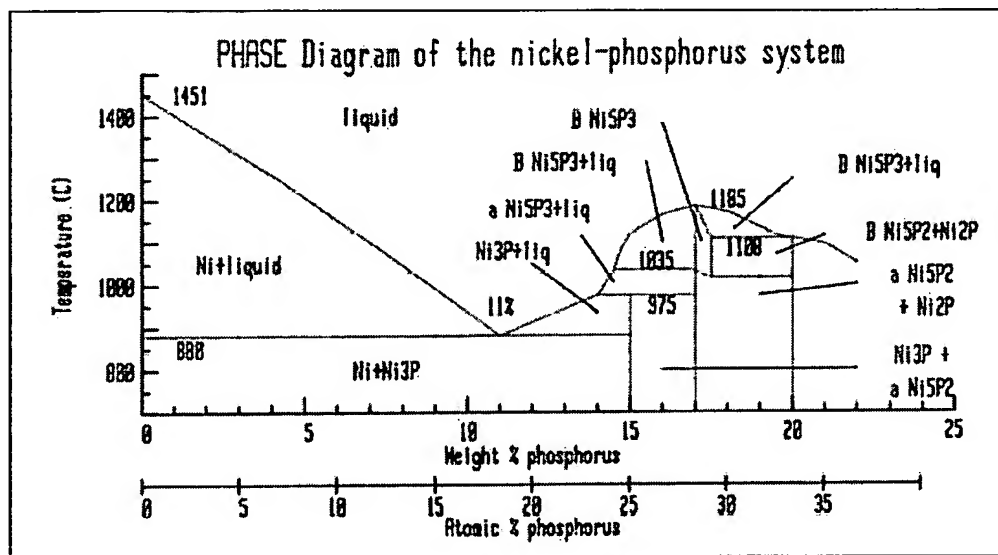

Figure 3 Phase Diagram

MECHANICAL PROPERTIES

HARDNESS

The hardness of electroless nickel deposits has been studied by several researchers and is quite well documented. The deposit hardness is measured by micro-sectioning 3 mil deposits and indenting with Knoop or Vickers diamond on the cross section. Loads of 25 to 1000 grams are used, however 100 grams is the norm. "Industrial Nickel Plating and Coating", INCO (1976).

The Hardness of the deposit is effected by the phosphorus and the structure. In the graph below the lower phosphorus content affords a higher as-plated hardness while the higher phosphorus affords a lower as-plated hardness.

HARDNESS of Electroless Nickel vs Temperature vs TYPE

Figure 4 Hardness
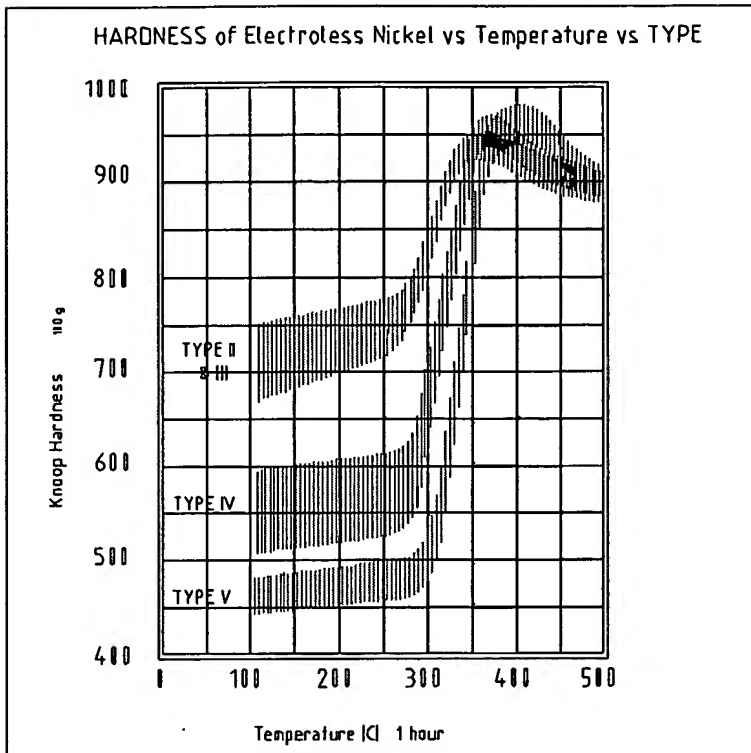
Vickers and Knoop diamond indenters have different indenting geometrics which will produce variations in hardness readings relative to orientation of the indenter face and lamellae. Using conversion tables to Rockwell C and other scales is not recommended and should only be used as an approximate.

The hardness can be increased by heating the deposit to above 320(C) and precipitating the nickel and phosphorus into a crystalline structure. Shorter times at higher temperatures will increase the precipitation.

MODULUS of ELASTICITY

The coating has a modulus of elasticity between 1.2 to 2 X 10^-4 Kp/mm2. The values are controlled by the phosphorus content and heat treatment. H. Wiegand, G. Heinke and K. Schwitzgebel reported that for 8% phosphorus alloys the Youngs Modulus increases with the rise in temperature for heat treatment Wiegand, H.; Heinke, G.; Schwitzgebel, K., "Metalloberfl", 22 (1968) 10, 304.

Electroless nickel deposits are not generally capable of much elongation (0.5% to 1.5%), and should not be applied when significant deformation of the substrate is required unless mechanical evaluation proves otherwise.

TENSILE STRENGTH

The tensile strength of the deposit is very important for many reasons. The range for 5% phosphorus is 40 +or- 5 Kp/mm2 for as-plated, and 80 + Kp/mm2 after 750(C) for two hour heat treatment. For 8.5% mass phosphorus within the deposit, the tensile strength is 70 Kp/mm2 for as-plated and 25 Kp/mm2 after 750(C) for two hours. Graham, A.H.; Lindsay, R.W.; Read, H.J., "Jnl Electrochemical Society", 109 (1963) 12, 1200, 112 (1965) 4, 401.

The control of the tensile strength appears to be the grain size and the internal stress, rather than the phosphorus. This is likely because having a larger number of defects will tend to concentrate the forces and cause mechanical failure.

The strength and elongation properties are in direct relationship to each other while hardness has an indirect relationship to strength.

During the 80s, additional work on hardness of low phosphorus deposits was performed. These studies revealed that relatively harder deposits were obtained when the alloy was very low in phosphorus.

In some applications this increased hardness is near equal to that of heat treated deposits. This property can be used when plating aluminum and other heat sensitive base materials.

After heat treatment at 750 (C) for 2 hours the deposit strength and hardness are significantly different. The grain size of Nickel phosphide will vary from between 10A to 150A depending phosphorus alloy and duration of heat treatment. As these Nickel phosphide crystals form the deposit shrinks and defects within the structure appear.
In the graph below, the deposits have been heated and precipitation is nearly complete. The strength of the deposit is a direct relation to the amount of shrinkage and phosphorus. In deposits of lower phosphorus alloy the strength increases providing for improved wear and mechanical service.

INTERNAL STRESS

The internal stress of the electroless nickel deposit is dependent upon the phosphorus content and the substrate. Typical values are between -3 MPa and 30 MPa on steel, with the lowest stress at 13% P. For most controlled low stress processes the internal stress can be maintained between +or- 2 MPa. Large variations and deviations in the normal curve are generally an indication of stress inducing trace elements, or of poor process control. These will cause the internal stress to become tensive (max 20 MPa). Parker, K., "Effects of Heat Treatment on the Properties of Electroless Nickel Deposits", PLATING AND SURFACE FINISHING, Dec. 1981, 73.; Gawrilov, G.G., "Chemical (Electroless) Nickel Plating", Portcullis Press, 1979, 43.; Parker, K., "The Stress of Electroless Nickel Deposit", AES Montreal, Qubec, June 25, 1970.

The trace elements of cadmium, bismuth, arsenic, antimony and silicon oils within the solution will cause the internal stress of the deposit to become more tensive.
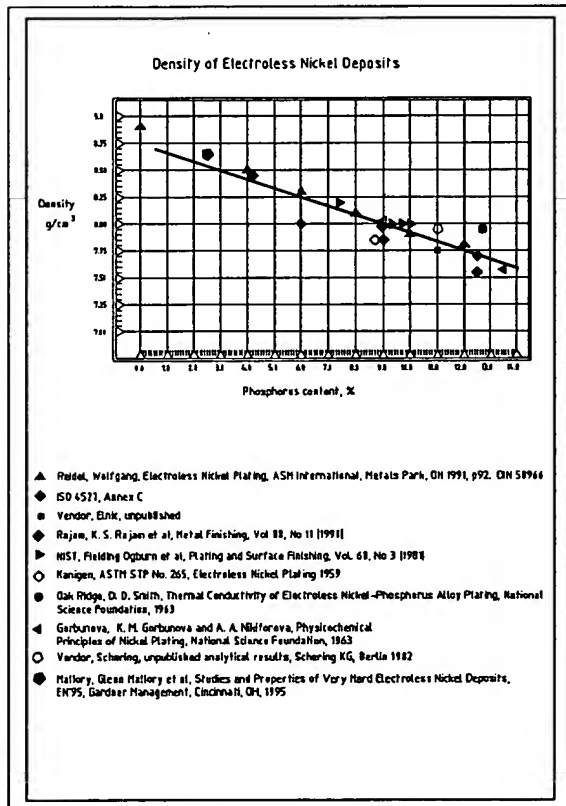
DENSITY

The density of electroless nickel deposits is lower than pure nickel due to the presence of phosphorus as an alloying constituent. The most common range of phosphorus present in commercially applied processes is 3% to 12%.

An electroless nickel deposit containing 3% phosphorus has a density of 8.52 g/cm3. An electroless nickel deposit with a 7.5% phosphorus content has a reported density of 7.92g/cm3. These values are lower than for pure metallurgical nickel of 8.91g/cm3. Krieg, A., "Symposium on Electroless Nickel Plating", ASTM Special Tech. publication no. 265 (1959) pg. 13. International Nickel, In 1996 a compilation of Density Analysis was made for the ASTM B733 Appendix.

Figure 5 Density

COEFFICIENT OF THERMAL EXPANSION / HEAT OF CONDUCTIVITY

The coefficient of thermal expansion of an 8% to 9% phosphorus content deposit is 13 to 14.5 X 10-6 in/in (C).

The heat of conductivity given for an electroless nickel deposit containing 8% to 9% phosphorus is calculated as 0.0105 - 0.0135 cal - cm/sec (C). Pure metallurgical nickel has a value of 0.198 cal - cm/sec (C). Gutzcit, G.; Mapp, E.T., "Kanigen Chemical Nickel Plating", Corrosion Technology, Volume 3, no. 10, Oct., 1956, London, pg. 331. In either case nickel is not a good thermal conductor, but when applied on an aluminum or copper substrate will provide an excellent system for thermal conductivity. This is primarily because corrosion products, which form over time on unprotected substrates, will isolate and reduce the net thermal conductivity of the system.

MELTING PROPERTIES

The melting range of electroless nickel deposits varies widely, depending upon the amount of phosphorus alloys in the deposit. The eutectic ,lowest melting point, is measured around 880(C) for commercially available processes with 3% to 12% phosphorus. This temperature corresponds to the melting point of NiP, nickel phosphide, which precipitated during heating of electroless nickel deposits.

The softening range is perhaps best represented by the X-Ray Analysis showing amorphous Nickel (1,1,1) where the deposits having around 11% by weight of phosphorus. A wide melting range indicates a low phosphorus content deposit with the upper limit eventually coinciding to that of pure metallurgical nickel (1450C). Krieg, A., "Symposium on Electroless Nickel Plating", ASTM Special Tech. Publication, No. 265 (1959) pg. 13.

Generally the alloy will remain under 11% phosphorus and the predominate structure will be Ni3P with a number of Ni clusters.

MAGNETIC PROPERTIES

Electroless nickel deposits which contain greater than 9% phosphorus are considered to be essentially non-magnetic as-plated. The coercivity of an 8.6% and 7.0% phosphorus content deposit were reported as 1.4 oersteds and 2.0 oersteds respectively. A 2.5% phosphorus content deposit produced a magnetic coating of 30 oersteds (as-plated). Brenner, A.; Couch, D.F.; and Williams E.K., "Electrodeposition of Alloys of Phosphorus and Nickel or Cobalt", Journal of Reaserch, NBS, Volume 44, (1950). When the phosphorus content was increased to 10%, the deposit was non-magnetic. In applications where the deposit must remain non-magnetic after heat treatment (275 (C) for one hour), it has been found that the formation of nickel clusters can be reduced significantly by processing.

Coating thickness measurements with devices which rely upon the non-magnetic characteristic of the coating may become inaccurate or require special calibration, if phosphorus contents are below 9%.

Heat treatment of electroless nickel will increase the ferromagnetic properties of the deposit. Deposits which contain above 9% phosphorus will become slightly magnetic when heat treated above 270 to 280 (C). At this temperature the solid solution of phosphorus in the deposit begins to form non-magnetic Ni3P, nickel phosphide, and magnetic nickel, Ni.

AUTONIC HX series process will produce a non-magnetic deposit for use as an undercoating for magnetic memory disks. Typical requirements for these coatings are that the deposit show no magnetic properties after a 280 (C) bake for 1 hour. This requires that the deposit is in the gamma state and that there are no co-deposited trace metals which initiate the precipitation to alpha nickel.

ELECTRICAL PROPERTIES

The electrical resitivity of pure metallurgical nickel has a value of 6.05 micro-ohms-cm. (1) Electroless nickel deposits containing 6 to 7% phosphorous have values (as plated) which range from 52 to 68 micro-ohms-cm. (2)m while a 13% phosphorous content deposit showed a significantly higher resitivity of 110 micro-ohms-cm.

Heat treating the electroless nickel coating reduces the electrical resitivity. Heat treatment up to 150 (C) produces changes in the deposit primarily attributed to structural averaging of the phosphorous content and liberation of absorbed hydrogen. In the range of 260 to 280 (C), heat treatment produces a further decrease in electrical resitivity. This change is attributed to the precipitation of Ni3P (Nickel Phosphide) in the coating. (1) An electroless nickel deposit with 7% phosphorous, heat-treated to 600 (C) was reportedly reduced from 72 to 20 micro-ohms-cm. International Nickel, "Engineering Properties of Electroless Nickel Deposits", 1971, pg. 5 This shift in conductivity is important in understanding the errors in measuring thickness when using an eddy current device.

POROSITY

Commercially applied electroless nickel deposits contain varying levels of porosity, since the amount and size of the pores will vary significantly from one deposit to another. Saubestre, Dr. E. B.; Hiden, Dr. Jaun, "Influence of Substrate Preparation on the Corrosion Resistance of Electroless Nickel Coatings", Enthone, Inc., 1968 Coating porosity can be a result of substrate porosity, surface conditions, surface properties, processing, and solution formulations.

Currently non-destructive porosity tests for metallic coatings (e.g. on steel) do not exist, such as those for non-metallic coatings. The major types of porosity tests for metallic coatings include direct application of a corroding solution which reacts with the coating surface (e.g. ferroxyl test). These methods are less practical for field inspection of components, especially if they are large parts and have recessed areas. Take note that porosity meters using mercury penetration are not generally capable of resolving pores smaller than 8 microns.

ADHESION

Excellent adhesion of electroless nickel deposits can be achieved on a wide range of substrates including steel, aluminum, and copper. Typical bond strengths reported for electroless nickel on iron and copper alloys range from 50 to 64 kpsi, Safranck, W.H., "Symposium on Electroless Nickel Plating", ASTM Special Technical Publication 265, 1959, pg. 41-49. The bond strength on light metals, such as aluminum, tends to be lower. For most alloys it is in the range of 15 to 35 kpsi. The forces which contribute to bond strengths are considered atomic in nature.

Heat treatment or baking is commonly employed to improve adhesion on all metals, particularly on light metals such as aluminum or titanium. During heat treatment a diffusion occurs between the close atoms of the coating and the substrate. The normal temperature range of heat treatment is 130 to 200 (C), from thirty minutes to several hours. Exposure of electroless nickel deposits to temperature above 218C) will begin to change the physical, mechanical, and protective properties of the electroless nickel deposit. This is due to the precipitation of nickel phosphide. Therefore, careful consideration should be given to the choice of heat treating conditions.

THICKNESS

Electroless nickel can be accurately and uniformly deposited in a wide range of coating thicknesses. It is accomplished on both small and large articles, and on components which may be fairly complex with recessed areas. Electroplating such parts would produce non-uniform deposition on ridges or sharp edges, and possible plating voids in more complex configurations. The thickness range for electroless nickel plating , for most commercial applications, is .05 to 10 mils Krieg, A., "Symposium of Electroless Nickel Plating", ASTM Special Technical Publication 2651, 1959, pg. 44-46. ,although heavier deposits have been produced. Coating as thick as 100 mills have been produced and 40 to 60 mill coatings are used as a replacement for hardchrome. Note: The primary thickness limitation is controlled by the ability of the process to remain chemically in balance, free of trace elements and excess solids. Coatings in excess of 100 mils have been accomplished with the use of excellent filtration and process control.

The majority of commercial applications, except those involving corrosive service or heavy build up on worn parts, utilize a thickness between 0.1 to 1.0 mils. Thicknesses of 1.0 to 3.0 mils are common for corrosive service, while deposit thicknesses above 3.0 mils are typical of repair and rework. Deposition of heavier coatings requires careful process control to avoid roughness and pitting.

ASTM B733 Specification provides a range of thicknesses as part of the classification and are described in the requirements of Service Condition. The selection of the correct thickness for each application will help assure that the desired performance is achieved.

Thickness measurements of electroless nickel deposits can be determined with magnetic devices, if the phosphorus content is above 9%. Below this level variations in the magnetic properties of the deposit affect the accuracy of this test method. The Beta Backscatter is accurate for thin deposits on aluminum (0.00001 to 0.005 in.) and for standards that have been prepared for density variation in the alloy.

Half masked steel transmission pins (centerless ground .250" x 1"), that have run with the article, can be read with a micrometer with reliable accuracy.

BRIGHTNESS

The brightness or reflectivity of electroless nickel deposits varies significantly, depending upon the specific formulations, the presence of brightening agents and the surface finish. An electroless nickel solution producing a bright deposit, may provide a dull deposit if the surface of the substrate is rough.

The use of specific trace metals in the formulation increase the number of latice defects in the deposit. These defects in turn increase reflectivity by displacing the normal distribution of nickel in the deposit. This condition is harmful to the corrosion resistance and mechanical properties of the deposit.

The appearance of electroless nickel deposits is similar to electro-deposited nickel, with slight yellowing due to oxidation. A slightly blue cast can be achieved by modifying the brightner trace metal.

TOXICITY

The possible injurious effects of orally ingested nickel deposits have been considered negligible. Hajdu, Dr. Juan B.; Yarkosk, Eugene; Marczak, Tony, "Corrosion Resistance of Electroless Nickel by Food Constituents", O-Han Inc.

While the toxicological and epidemiological effects of nickel have not been completely established, lately several researchers have been studying the carcinogenic effects of nickel and have reported a potential problem. Nickel sub-sulfide dust appear to be the root cause of the problem. These forms of nickel are not available in the plating operations but good industrial hygiene would recommend that the operators be protected from mist and vapors.

Within the EEC a stringent control of nickel metal have been considered. The regulation of air borne nickel has been discussed in recent years and a slow effort by regulators has been underway.

CORROSION RESISTANCE

The corrosion resistance of the electroless nickel deposit has already been of great interest to engineers and designers. There are several reasons for using electroless nickel in corrosion applications. These include: 1) protection of the substrate. 2) maintain the precision of the article. 3) protection of the product from contamination.

Encapsulation is the principle method of protection and corrosion control of the substrate. This is afforded by the electroless nickel plating solution penetrating into the small voids and surface imperfections, as well as on the surface, chemically reducing a finely divided nickel (1,1,1) and phosphorus alloy.

Electroless nickel phosphorus deposits have been used in a number of environments with success. These include alkalis, salts and brine solutions, petroleum, sweet and sour natural gas applications, solvents and hydrocarbons. Other environments which can be considered for intermittent use are organic acids, reducing inorganic acids and strong oxidizing media. In these environments, the protection of the coating is provided by an oxide film which forms a passive layer, reducing the galvanic currents between grain boundaries. The removal or disruption of this film may dramatically increase the corrosion rate of the deposit.

To accurately develop an understanding of the pitting qualities of a deposit, all of these factors must be evaluated simultaneously. Many accelerated tests have been developed to evaluate the potential and presence of pits in the deposit.

The use of porosity tests can become an important tool in quality control, if they are in fact used in quality control. Continuous monitoring and evaluation of all process constraints will provide the plater with the information necessary to maintain the pore-free desired condition on all substrates. Faulty quality control programs will produce erroneous results and misinformation.

The coating itself is very corrosion resistant in many environments. The major factors controlling this deposit quality are 1) the surface condition, 2) alloy composition and physical properties, 3) structural integrity and 4) the nature and composition of the environment. Like porosity, each of these factors must be analyzed simultaneously for a quality control program to be effective.

The use of immersion tests can be of value when evaluating the composition and nature of the service environment for the deposit. Other factors, like erosion or oxidation of the deposit in dynamic conditions, will alter the performance.

Additional tests are required to analyze the alloy composition. These are executed by measuring variables in the solution's composition and the process constraints. These variations will control the daily performance of the deposit and are far more relevant to the minimum corrosion performance desired by the purchaser.

To insure a consistent deposit important constraints, such as temperature, pH, nickel and hypophosphite content, and trace metals present should be monitored on a daily basis as

part of a comprehensive corrosion quality assurance program. Wide fluctuations in the nickel and phosphorus alloy, as well as iron, chrome, and stress inducing metals, will affect how the electroless nickel deposit will perform.

Immersion tests will evaluate the corrosion resistance of the coating. The weight loss indicates what the general corrosive nature of the environment is upon the deposit. It can serve as an engineering guide for application and development. A partial listing of environments and corrosion rates are provided in TABLE 2. Metzger, W.H., "Symposium of Electroless Nickel Plating", ASTM Special Technical Publication 265, 1959, pg. 18-19.; Duncan, R., "AES Electroless Nickel Symposium", March 1982. Additionally, electrochemical tests can be used to produce corrosion rates and porosity values which represent a practical means of generating accurate and fast corrosion data. The EC test can be operated at temperatures in excess of 200(C) and pressures greater than 1000 psi.

Immersion of electrochemical analysis may not always be sensitive to the aggressive nature of the applications, and should only be used as a quality control too or guide. Actual service should be the basis of evaluation for corrosion application development.

CORROSION SUMMARY

Electroless nickel deposits significantly control corrosion and protect products over a wide range of conditions in a variety of environments. Existent today are quality control tools to evaluate the process and deposit, insuring consistent performance and customer satisfaction.

Table 2 has been prepared from tests performed by Stapleton Technologies and from published information from studies on electroless nickel over the last thirty years. This information can be used to provide an approximate rate of corrosion and can be used to guide your selection of electroless nickel coatings.

Electrochemical and exposure testing is available at Stapleton Technologies for evaluation of environments which are not listed in table 2. Contact Stapleton Technologies for more information.

KEY TO TABLE 2	
EC	ELECTROCHEMICAL
WT	WEIGHT LOSS
MPY	MIL PER YEAR PENETRATION
DEPOSITS:	A (NiP8)
	B (NiP11)

Figure 6 Chemical Processing

CORROSION RESISTANCE OF ELECTROLESS NICKEL, TABLE 2

CORROSIVE AGENT	TEMP. (C)	IMMERSION	AERATION	TEST TYPE	RATE MPY	DEPOSIT
Acetic Acid 5%	22	total	NA	4 weeks WT	0.791	A
Acetic Acid 5%	22	total	0^2	4 weeks WT	5.950	A
Acetic Acid 5%	22	total	0^2	20 weeks WT	0.845	A
Acetic Acid 10%	22	total	NA	16 weeks WT	0.181	A
Acetic Acid 10%	22	total	0^2	8 weeks WT	1.131	A
Acetic Acid 50%	22	total	NA	20 weeks WT	0.165	A
Acetic Acid 50%	22	total	0^2	8 weeks WT		
Acetic Acid, Glacial	22	total	NA	20 weeks WT	0.019	A
Acetic Acid, Glacial	22	total	0^2	8 weeks WT	0.990	A
Acetic Acid, Glacial	20	total	NA	8 weeks WT	0.80	B
Acetone	22	total	NA	16 weeks WT	0.003	A
Acetone	20	total	NA	8 weeks WT	0.08	B
Aluminum Sulfate, 27%	22	total	NA	8 weeks WT	5.00	B
Ammonia, 25%	20	total	NA	8 weeks WT	16.00	B
Ammoniated Ammonium:						
Nitrate	22	total	NA	16 weeks WT	0.310	A
Nitrate	22	total	NA	12 weeks WT	0.31	A
Nitrate Vapor	22	total	NA	16 weeks WT	0.258	A
Nitrate Vapor	22	total	NA	16 weeks WT	0.28	A
Ammonium: Chloride,						
saturated 30% NH^3	22	total	NA	16 weeks WT	0.230	A
Hydroxide 30% NH^3	22	total	NA	16 weeks WT	2.30	A
Hydroxide 30% NH^3	22	total	NA	29 weeks WT	0.062	A
Hydroxide 30% NH^3	22	partial	NA	24 weeks WT	0.158	A
Nitrate 20%	20	total	NA	8 weeks WT	15.00	B
Nitrate 63%	22	total	NA	28 weeks WT	0.0135	A
Nitrate 63%	22	total	NA	24 weeks WT	0.0103	A
Phosphate 5%	22	total	NA	4 weeks WT	0.71	A
Sulfate saturated	22	total	NA	20 weeks WT	0.0499	A
Sulfate saturated	22	total	0^2	20 weeks WT	0.0169	A
Sulfate saturated	20	total	NA	8 weeks WT	3.00	B
Sulfate 5%	22	total	0^2	8 weeks WT	1.19	A
Sulfate 5%	22	total	0^2	20 weeks WT	0.090	A
Sulfite saturated	22	total	NA	20 weeks WT	0.016	A
Thiocyanate	22	total	NA	20 weeks WT	0.171	A
Amyl Acetate	22	total	NA	24 weeks WT	0.002	A
Amyl Alcohol	22	total	NA	24 weeks WT	none	A
Amyl Chloride	22	total	NA	24 weeks WT	0.013	A
Aniline Hydrochloride saturated	22	total	NA	16 weeks WT	0.468	A
Beer	22	total	NA	4 weeks WT	0.217	A
Benzene	20	total	NA	8 weeks WT	nil	A-B

CORROSION RESISTANCE OF ELECTROLESS NICKEL, TABLE 2

CORROSIVE AGENT	TEMP. (C)	IMMERSION	AERATION	TEST TYPE	RATE MPY	DEPOSIT
Benzyl Acetate	22	total	NA	24 weeks WT	none	A
Benzyl Alcohol	22	total	NA	28 weeks WT	0.004	A
Benzyl Chloride	22	total	NA	16 weeks WT		
Black Liquor Skimmings	22	total	NA	12 weeks WT	0.010	A
Boraxo saturated	22	total	NA	32 weeks WT	0.120	A
Boric Acid	22	total	NA	8 weeks WT	0.527	A
Brine, 3.5% salt CO_2 saturated	95	total	NA	8 weeks WT	5.00	B
Brine, 3.5% salt H2S saturated	95	total	NA	8 weeks WT	nil	B
Calcium Chloride:						
42%	20	total	NA	8 weeks WT	0.20	B
48.5%	22	total	NA	32 weeks WT	0.008	A
48.5%	22	total	0_2	32 weeks WT	0.040	A
48.5%	22	total	NA	32 weeks WT	0.001	A
48.5%	22	total	0_2	30 weeks WT	0.011	A
Carbon Disulphide	22	total	NA	21 weeks WT	none	A
Carbon Disulphide and Water	22	total	NA	21 weeks WT	none	A
Carbon Tetrachloride	22	total	NA	32 weeks WT	0.005	A
Carbon Tetrachloride	20	total	NA	8 weeks WT	none	B
Cetyl Alcohol, molten	71	total	NA	8 weeks WT	0.008	A
Citric Acid 5%	22	total	NA	16 weeks WT	0.033	A
Citric Acid 5%	22	total	0_2	8 weeks WT	0.0744	A
Citric Acid saturated	20	total	NA	8 weeks WT	7.00	B
Cobalt Linoleate	22	total	NA	16 weeks WT	none	A
Cresylic Acid	22	total	NA	16 weeks WT	0.002	A
Cupric Chloride, 5%	20	total	NA	8 weeks WT	25.00	B
Detergent solution 5% TIDE	22	total	NA	4 weeks WT	0.037	A
Dibutyl Phthalate	22	total	NA	8 weeks WT	0.006	A
Diphenyl, molten	71	total	NA	8 weeks WT	0.009	A
Ethylene Glycol (STANDARD CORROSION TEST)				3 weeks WT	0.0255	A
Ethylene Glycol	20	total	NA	8 weeks WT	0.600	B
Ferric Chloride, 1%	20	total	NA	8 weeks WT	200.0	B
Fluorphosphoric Acid	22	total	NA	12 days WT	1.560	A
Formaldehyde, 37%	22	total	NA	28 weeks WT	0.007	A
Formaldehyde, 37%	22	partial	NA	24 weeks WT	0.158	A
Formic Acid, 88%	20	total	NA	8 weeks WT	13.00	B
Gasoline	22	total	NA	4 weeks WT	0.022	A
Glucose	22	total	NA	16 weeks WT	none	A
Hydrochloric Acid, 5%	20	total	NA	8 weeks WT	24.00	B

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CORROSION RESISTANCE OF ELECTROLESS NICKEL, TABLE 2

CORROSIVE AGENT	TEMP. (C)	IMMERSION	TEST AERATION	RATE TYPE	MPY	DEPOSIT
HCL solution:						
pH 1.5	22	total	NA	4 weeks WT	1.250	A
pH 1.5	22	total	0^2	4 weeks WT	5.3207	A
pH 1.5	22	total	0^2	8 weeks WT	3.050	A
pH 2.0	22	total	NA	4 weeks WT	0.931	A
pH 2.0	22	total	0^2	12 weeks WT	2.500	A
pH 2.0	22	total	02	8 weeks WT	0.630	A
pH 2.5	22	total	NA	4 weeks WT	0.33	A
pH 2.5	22	total	0^2	12 weeks WT	0.638	A
pH 3.0	22	total	NA	4 weeks WT	0.14	A
pH 3.0	22	total	0^2	12 weeks WT	0.168	A
pH 3.5	22	total	NA	4 weeks WT	0.074	A
pH 3.5	22	total	0^2	4 weeks WT	0.410	A
pH 3.5	22	total	0^2	32 weeks WT	0.0092	A
pH 4.0	22	total	NA	4 weeks WT	0.054	A
pH 4.0	22	total	0^2	10 weeks WT	0.125	A
pH 4.0	22	total	0^2	32 weeks WT	0.015	A
Hydrofluoric Acid, 2%	20	total	NA	8 weeks WT	27.00	B
Insectisol	22	total	NA	3 weeks WT	0.008	A
Isoamyloctyl						
Orthophosphate:75%	22	total	NA	3 weeks WT	none	A
Lactic Acid:						
45%	22	total	NA	16 weeks WT	0.096	A
45%	22	total	0^2	4 weeks WT	1.566	A
45%	22	total	NA	32 weeks WT	0.009	A
45%	22	total	0^2	16 weeks WT	0.822	A
80%	22	total	NA	32 weeks WT	0.052	A
80%	22	total	0^2	8 weeks WT	0.704	A
80%	22	total	NA	32 weeks WT	0.006	A
80%	22	total	0^2	16 weeks WT	0.145	A
85%	20	total	NA	8 weeks WT	1.00	B
Lead Acetate, 36%	20	total	NA	8 weeks WT	0.20	B
Lemon Juice:						
Canned	22	total	NA	4 weeks WT	0.230	A
+0.1% Na Benzoate	22	total	NA	23 DAYS	0.910	A
Methyl Alcohol	22	total	NA	24 weeks WT	none	A
Naphtha, odorless	22	total	NA	3 weeks WT	none	A
Nitric Acid, 1%	20	total	NA	8 weeks WT	3.00	B
Oleic Acidride	22	total	NA	10 weeks WT	0.012	A
Orange Juice, canned	22	total	NA	4 weeks WT	0.013	A
Oxalic Acid, 10%	20	total	NA	8 weeks WT	3.00	B
Petroleum, sour crude	22	total	NA	2 weeks WT	0.001	A
Petroleum, white oil	22	total	NA	3 weeks WT	none	A
Phenol, 90%	20	total	NA	8 weeks WT	0.20	B
Phosphoric Acid, 85%	22	total	NA	28 weeks WT	0.008	A
Phosphoric Acid, 85%	20	total	NA	8 weeks WT	3.00	B

CORROSION RESISTANCE OF ELECTROLESS NICKEL, TABLE 2

CORROSIVE AGENT	TEMP. (C)	IMMERSION	AERATION	TEST TYPE	RATE MPY	DEPOSIT
Photographic:						
Developer	22	total	NA	4 weeks WT	none	A
"Hypo" solution	22	total	NA	4 weeks WT	none	A
Refinery Brine solution	22	total	NA	3 weeks WT	none	A
Potassium						
Hydroxide, 50%	20	total	NA	8 weeks WT	none	B
Rosin Size:						
Concentrated (90 reflux)		total	NA	3 weeks WT	0.008	A
50% (90 reflux)		total	NA	3 weeks WT	0.058	A
Sodium:						
Carbonate, 10%	22	total	NA	4 weeks WT	none	A
Carbonate, saturate	20	total	NA	8 weeks WT	1.00	B
Cyanide, 5%	22	total	NA	4 weeks WT	0.520	A
Hydrosulfide, 40%	22	total	0^2	20 weeks WT	slight wt gain	A
Hydrosulfide, 40%	22	total	0^2	24 weeks WT	slight wt gain	A
Hydroxide, 10%	22	total	NA	4 weeks WT	none	A
Hydroxide, 50%	95	total	NA	8 weeks WT	0.20	B
Hydroxide, 72% (116 reflux)		total	NA	16 weeks WT	0.069	A
Hydroxide, 72% (116 reflux)		total	NA	24 weeks WT	0.362	A
Sulfate, 10%	20	total	NA	8 weeks WT	0.80	B
Steric Acid, 5%	70	total	NA	4 weeks WT	0.02	A
Sulfuric Acid: 1%	22	total	NA	4 weeks WT	1.120	A
1%	22	total	0^2	8 weeks WT	1.550	A
5%	22	total	NA	1 week WT	1.190	A
5%	22	total	NA	16 weeks WT	0.442	A
5%	22	total	0^2	4 weeks WT	1.7340	A
65%	20	total	NA	8 weeks WT	9.00	B
Tall Oil, crude	22	total	NA	12 weeks WT	0.31	A
Tall Oil, refined	22	total	NA	32 weeks WT	0.017	A
Tanning sol. (Korean)	22	total	NA	3 weeks WT	0.048	A
Thionyl Cl, anhydrous	22	total	NA	3 weeks WT	0.032	A
Urea, 25%	22	total	0^2	16 weeks WT	0.045	A
Urea, 25%	22	total	0^2	16 weeks WT	0.009	A
Urea, saturated	22	total	0^2	16 weeks WT	0.046	A
Urea, saturated	22	total	0^2	16 weeks WT	0.008	A
Water, Acid Mine, 3.3 pH	20	total	NA	8 weeks WT	7.00	B
Water, Chlorine:						
5 ppm Cl^2	22	total	NA	4 weeks WT	0.018	A
10 ppm Cl^2	22	total	NA	4 weeks WT	0.012	A
Water, Deionized	22	total	0^2	12 weeks WT	0.020	A
Water, Deionized	49 reflux	total	NA	12 weeks WT	0.012	A
Water, Deionized	82 reflux	total	NA	12 weeks WT	0.14	B

Table 2. Chemical Resistance of Electroless Nickel in Environments

WEAR PROPERTIES

This section covers the wear mechanism and mechanical testing of wear, for the evaluation of the electroless nickel deposit.

Electroless nickel deposits have been used in wear service for over thirty five years. Typical service conditions include, pump housings, ball valves, print rollers, hydraulic cylinders, electronic parts, connectors, memory disks, artillery parts and myriad other applications.

Plating with electroless nickel affords flexibility when attempting to protect a base material (substrate), with a thin, hard, amorphous, nickel coating. The qualities of corrosion resistance, uniformity, lubricity, hardness and low stress, combine to provide an excellent bearing and wear surface. This technology can be exploited to solve a vast array of specific problems.

Wear is the mechanical loss of material on a surface. There are many types of wear and nearly two hundred specific wear tests available. This affords a complex evaluation of any electroless nickel application. We have divided wear into four categories, which can be combined to define most wear situations.

FRETTING WEAR

Fretting wear is produced when the surface is mechanically deformed, causing material to be fractured and removed. A recent application which is required electroless nickel to eliminate fretting was Diesel valve spring retainers on locomotive engines. As the springs and valve system moved, the retainer clips, made of hardened RC 35 steel, exhibited fretting, causing movement and wear debris on the valve stem. To eliminate this fretting, .0003 inches of electroless nickel was applied with a 375 (F) 1 hour bake for hydrogen embrittlement release. Now the retainers do not show any sign of fretting wear at overhaul.

Fretting wear generally occurs when there ia small movement at high loads. This type of wear can be simulated by ball and block or pin and notch block with loads in excess of 1000 lbs. with oscillatory motion.

The development of a standard test method for fretting wear has not been proposed, nor is there any reference of individuals testing for fretting of this deposit. This area requires further study and should be a subject of further investigation.

ADHESION WEAR

Adhesion wear is the most common in metal-to-metal wear situations. It is produced when microwelds cause the material interface to join, and the adhesion failure of the coating or wearing material occurs below the weld. The structure of the alloy and morphology of the deposit is important in determining the degree of the adhesion failure and the wear performance of the coating.

This type of wear is produced on cam journals and high load surfaces of similar materials. In these applications, and lubrication failure will generally cause immediate catastrophic

failure of the bearing surface. For many years, electroless nickel has been providing the lubricity and hardness at the interface to significantly reduce the adhesion wear and improve the service of the plated article.

Adhesion wear can be evaluated by using ring and block type test LFW-1, run lubricated or dry. Analysis of the friction and wear scars with weight loss correlation can be used to describe the dynamics of the mating surfaces.

There are several ways to describe adhesion wear; weight loss of ring and block, wear wear scars length and depths, volume of wear debris relative to coefficient of friction, hardness, and heat treatment. Other important factors include the type of lubricant used, load, surface finish, speed, and the acceleration at the start.

Many studies have been performed on electroless nickel deposits for adhesion wear. Measurements are generally taken on the scar length due to the accuracy of the measurement, rather than the change of radii.

The correlation of weight loss, density measurements, and scar dimension make the assumption that the scar is uniform and that wear debris does not build on the trailing edge.

In most experiments, profilometer analysis have been run at regular intervals. This data can then be used to compute the exact volume of material lost, and produce a more accurate result.Tucker, R.C.; Miller, A.E., "Low Stress Abrasion and Adhesion Wear Testing", ASTM STP 615 pg. 76-89

Wear testing of electroless nickel on a continuous basis using the LFW-1 design by the NIST (National Institute of Standards and Technology) shows that a 5% P 890 (HK 500) electroless nickel (HAC formulation) wears at a rate of 0.07×10^{-4} mm3/m, while a chrome deposit with a hardness of HK 500 wears at a rate of 0.18×10^{-4} mm3/m.

Note: These units take into effect the linear wear for the duration of the test. The tests were run dry and revealed a coefficient of friction of 0.77 for the electroless nickel against electroless nickel while hardchrome against hardchrome was 1.02. Ruff, A.W.; Lashmore, D.S., "Dry Sliding Wear Studies of NiP and Cr Coatings on 02 Tool Steel" G2 Wear Symposium, Phoenix AZ Dec. (1981)

These results provide some basis for using electroless nickel as a barrier on one surface providing separation between steels.

Other LFW-1 tests confirm that the minimum wear lowest coefficient of friction is obtained with coatings which have a hardness of 890 (HV100). With lubricated plated blocks 890 (HV100) 540C/1, wear is about 2 mg/5000 cycles, while ring wear is 0.7 mg. With lubricated plated blocks 890 (HV100), the ring wear is negligible with wear debris building up on the block. In these tests, electroless nickel is running against steel with a final coefficient of friction for as-plated and heat treated 0.12. Parker, K., "Hardness and Wear Resistance Test of Electroless Nickel Deposits", AES Technical Conference, Chicago, June, 1974

When precipitation continues to recrystalize, the wear on the ring will increase to 5 to 10 mg/25000 cycles and the block will still collect debris with minimal wear.

Important constraints in evaluations are the speed of the ring, the loading force on the block, the type of lucricant, temperature of the test, surface condition, and the starting condition.

ABRASION WEAR

Abrasion wear is the result of solids being cut out of the surface. It is produced when paper, textiles, debris, ect. pass over the wearing surface and cut into the substrate, removing material.

Electroless nickel has been applied to print rollers, copier feed mechanisms, textile equipment, and many other similar applications to significantly to reduce the abrasive wear and extend the service of the plated article.

Abrasive wear can be evaluated by abrasive wheels that run over a coated test panel without lubrication. This test can be useful in evaluating the general abrasion performance of materials.

A recent-round robin with ASTM B08.08 has produced a test method which has reasonable laboratory correlation. Using a Taber Abrader with CS10 wheels and redressing the wheels after each 1000 cycles for 50 cycles and analyzing for weight loss, reasonable data was produced between three labs while the forth was low.

The reason for the variations, especially on the as-plated deposit wear, has been discussed by those working on the project. It has been assumed that the lower humidity in the laboratory in Tucson and the subsequent increase in the static charge on the wheel produced the variation. It is presumed that the vacuum was unable to remove wear debris, on the wheels producing a bearing, making the appearance of less abrasive wear.

Typical abrasive wear values for electroless nickel range from between 2.5 mg/1000 cycles to 30 mg/1000 cycles. Hardness and deposit morphology are important in the abrasion wear mechanism, with a 5% P, 980 (HV100), 400 C/1 hr. deposit wearing at 9 mg/1000 cycles and as-plated 5% P wearing at 18 mg/1000 cycles. Obtaining very low abrasion wear values (less than 4 mg/1000 cycles) is accomplished by heat treating for one hour in excess of 650 (C).

EROSION WEAR

Erosion wear is the most complex of the four varieties of wear. It involves the movement of liquids, solids, and gasses near to or on the surface. This can produce micro failure and abrasion. It eventually causes macro amounts of material to be removed. In order to better describe the erosion phenomenon, we will break this subject down into different mechanical phases.

SINGLE PHASE EROSION

This is the type of erosion where a liquid or gas is directed toward a surface, with the eroding material being free of all other phases. A liquid flow in an elbow or pipe, which is solid and gas free, or a gas flow in an elbow or pipe, void of solid and liquid, would be a single phase wear system. In actual service this type of wear phase may be impossible to obtain.

MULTI PHASE: LIQUID-GAS

This type of wear can be tested by degassing and filtering a neutral solution of deionized water and directing it through a venturi and across a surface. A venturi device provides the means of converting kinetic head into static pressure, causing the vapor pressure to be equal to the static pressure, which causes cavitation. This test represents a means of modeling the general flow regimens of a hydraulic system. Constraints which control this model include flow rate, temperature, head pressure and venturi dynamics.

The mechanism of wear for this test is produced by the formation of cavitation bubbles ahead of the specimen, and their subsequent decay and micro-implosion, causing removal of the electroless nickel deposit. Since the surface of an electroless nickel deposit is protected by an oxide film, cavitation is far more critical in corrosion services.

Results from the venturi testing shows that regions along the erosion specimen receive various degrees of wear based upon vortex and static pressure drop across the surface. Microscopic examination of the specimen can be used to evaluate the location and depth of the erosion wear.

MULTI PHASE:

SOLID, LIQUID AND GAS WEAR

This erosion wear is more complex, and the development of a standard test does not seem likely. Constraints, like solid particle size and structure, velocity and impingement angle and the corrosive nature of the liquid, control the total erosion process.

The venturi device used to test liquid-gas systems can also be used to test for solid-liquid-gas wear. Sand of 200 um to 300 um diameter is slurried at 20 gm/min into the flow, which is directed towards the specimen. Wear is often evaluated by microscopic examination for location and depth of wear for given velocity and impingement angle.

WEAR SUMMARY

Electroless nickel deposits will continue to find new wear applications. The development of standard test methods for plated coatings, especially electroless nickel, will require the industry to classify the main types of wear. The Taber abrasion test method has been adopted by ASTM in lieu of other, more reliable, technology. The Taber abrasion method has a reliability factor of around forty per cent. The need for better formulations is implicit, and specifically relating to electroless nickel, other wear tests will be required before more exacting comparisons between electroless nickel deposits can be made.

Wear testing is a good means of classifying wear performance between electroless nickel and other coatings subjected to the same tests. Actual performance is dependent upon many factors. A laboratory model, in some instances, is not able to predict the true wear found in a specific application. Periodic inspection of the wearing surface is recommended, as is documentation of case histories.
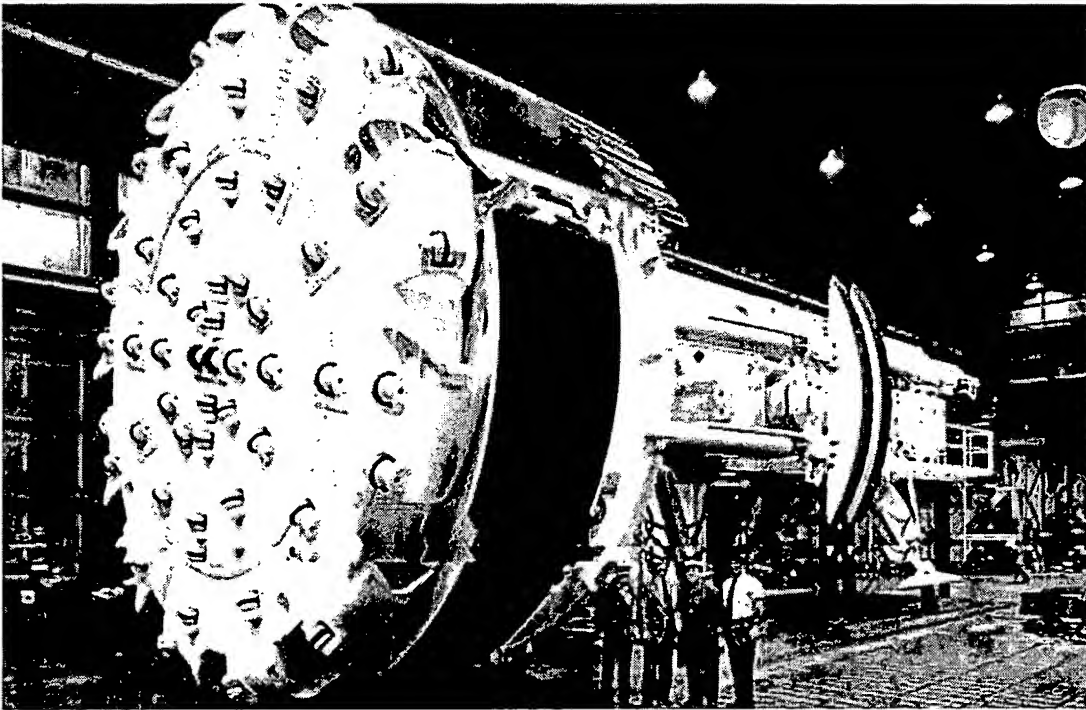

Figure 7   Horizontal Tunneling Tool, Ebco Industries Inc.